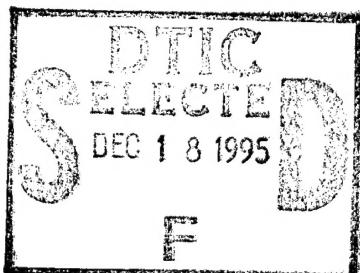


# Residual Stress and High-Temperature Creep Behavior in Carbon-Carbon Composites



Prepared by  
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30 September 1987

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## ABSTRACT

Residual stress is measured in a carbon-carbon composite. The curvature of a slice of asymmetric cross section indicates the amount of stress caused by thermal expansion anisotropy. Measured high-temperature creep rates are used to estimate stress relaxation times at high temperatures.

#### ACKNOWLEDGMENTS

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## CONTENTS

ABSTRACT.....	v
ACKNOWLEDGMENTS.....	vii
I. INTRODUCTION.....	1
II. EXPERIMENTAL.....	3
REFERENCES.....	7

## I. INTRODUCTION

Fabrication of carbon-carbon (C-C) composites requires high-temperature processing of carbon fibers with a carbonaceous matrix precursor. Residual stresses develop from the large thermal expansion anisotropy in graphite<sup>1</sup> and from the large variability in the local preferred orientation of the material, owing to the arrangement of fibers and matrix.<sup>2</sup> Such stresses frequently cause damage during the processing cycle,<sup>3</sup> but the high temperatures involved make it difficult to determine the internal stresses directly.

However, evidence for residual stress can be obtained from measurements at normal temperatures, such as those made in studying cracking in processed cylindrical carbon-carbon composites,<sup>3</sup> and in single carbon filaments.<sup>4</sup> Reported here are sample measurements of residual stress in 3D C-C composites, from which approximate values of residual strain were calculated. Stress relaxation behavior was estimated from calculations based on data obtained from high-temperature creep measurements on impregnated C-C yarns.

## II. EXPERIMENTAL

Three thin slices of differing thicknesses were excised parallel to the x-direction with a diamond saw from processed Cartesian weave 3D C-C composite billets. The unit-cell dimensions of the composite are shown in Fig. 1.

The effect of residual stress in the composite is manifested by the warping of the 0.25-mm slice (Fig. 2): One face has a substantial volume fraction of fibers running parallel to the long direction; the opposite face presents almost none. The sample, then, can be treated as a composite beam of asymmetric cross section. Given that the thermal expansion coefficient is smaller in the fiber direction than perpendicular to it, and that the face of the beam opposite the longitudinally reinforced face has mostly transversely oriented fibers and matrix, that face will have a greater thermal expansion. Qualitatively, that expansion should lead to greater contraction of the transverse face than the longitudinal face when cooling from a nominally stress-free temperature during processing, and should cause the transverse face to be concave, which is borne out by the evidence in Fig. 2. The probable reason why the other samples show much less curvature is that they are thicker and contain more longitudinal fibers on both faces, being  $\gtrsim 1$  unit cell in thickness.

To obtain a quantitative, albeit crude, estimate of residual strain, we compare the sample to a similar, homogeneous beam and apply simple beam theory, neglecting the effect of the asymmetric cross section and the nonuniform modulus and thermal expansion across the beam. (A more rigorous analysis has been performed by Jortner.<sup>6</sup>) The maximum strain from simple beam theory is given by

$$\epsilon_{\max} = \frac{1}{2} \left( \frac{t}{R} \right) \quad (1)$$

where  $t$  is thickness and  $R$  is radius of curvature. From Fig. 2,  $R$  is about 106 mm for the 0.25-mm-thick sample, giving  $\epsilon_{\max} = 1.2 \times 10^{-3}$ , less than the typical maximum strain to failure observed in the bulk material.<sup>5</sup>

Change in the residual stress can be observed by the change in curvature in the sample as it is heated,<sup>7</sup> further confirming that the curvature is

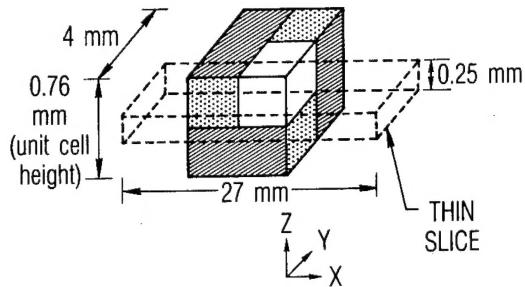


Fig. 1. Geometry of unit cell and thin slice along x-direction; 2-2-3 weave construction contains 2, 2, and 3 yarns per site in the x-, y-, and z-directions. 3D yarn preform, PAN-type, 380 GPa (55 Msi), pitch impregnation and densification.<sup>5</sup>

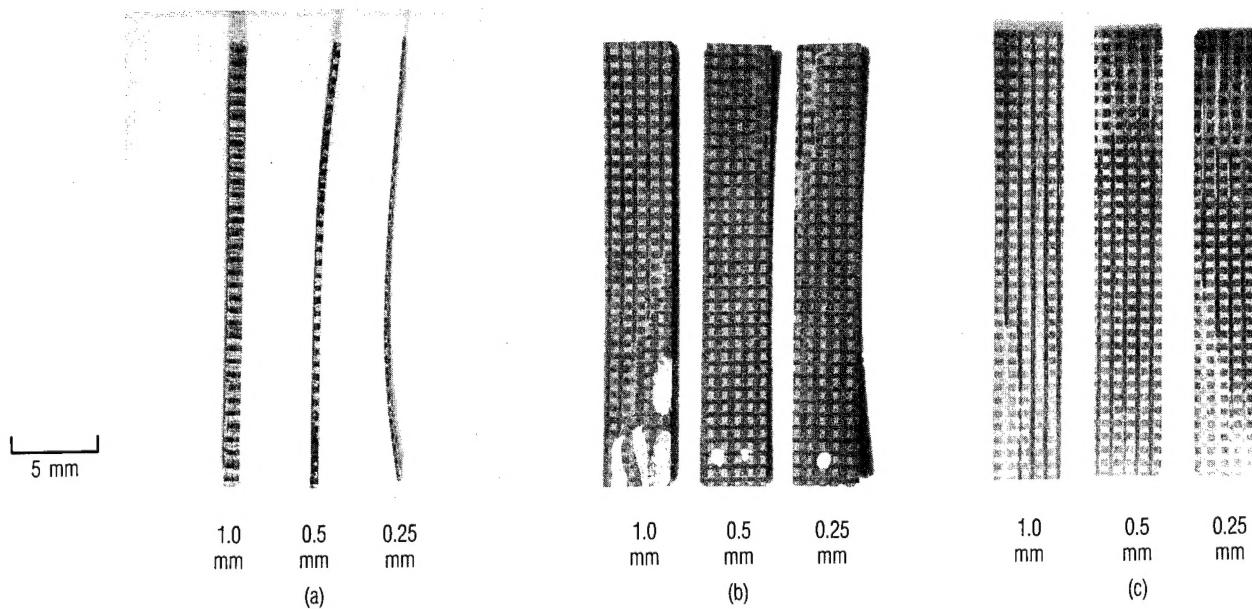


Fig. 2. X-direction slices of 2-2-3 composite: (a) cross-sectional view; (b) concave side; and (c) convex side.

caused by residual stress, because the curvature changes as the strains in the different components change on heating, from thermal expansion.

Characteristic stress relaxation times can be estimated from the measurements from

$$t_{\text{char}} = \frac{\epsilon_{\text{residual}}}{\dot{\epsilon}(T)} \quad (2)$$

in terms of the initial residual strain and creep rate. Creep rate at constant stress is assumed to be given by<sup>8</sup>

$$\dot{\epsilon}(T) = \dot{\epsilon}_0 \exp\left(\frac{-E}{kT}\right) \quad (3)$$

where  $\dot{\epsilon}_0$  is a material constant, k is Boltzmann's constant, and T is absolute temperature. As an example, we assume a maximum residual stress of  $1 \times 10^{-4}$ . Strain rates as a function of temperature have been measured on unidirectional composites of WCA rayon-based cloth yarn (Amoco Performance Products), with A240 pitch-based matrix, by high-temperature creep measurements.<sup>8</sup> Creep rates over the range 2400-2800°C at 17.2 MPa (2.5 ksi) fit to Eq. (3) give  $E = 451$  kJ/mole (108 kcal/mole) and  $\dot{\epsilon}_0 = 4.53 \times 10^2$ /sec. The typical macroscopic Young's modulus, 57 GPa (8.3 Ms), gives a maximum residual stress of 5.7 MPa (830 psi), which is a factor of ~ 3 smaller than the experimental stress. Thus, the estimated relaxation times are minima.

Listed in Table 1 are values of characteristic relaxation times at different temperatures calculated from these assumptions. Judging from fits to the data from high-temperature creep measurements, stress relaxation should proceed relatively rapidly at high temperatures (2500°C and above), and should begin to take exceptionally long as the temperature falls below 2000°C.

Table 1. Relaxation Times versus Temperature

T(°C)	Creep Rate (sec <sup>-1</sup> )	Relaxation Time
3000	$2.8 \times 10^{-5}$	3.6 sec
2500	$1.4 \times 10^{-6}$	1.2 min
2000	$1.9 \times 10^{-8}$	1.5 hr
1500	$2.2 \times 10^{-11}$	52 days
1000	$1.3 \times 10^{-16}$	24,000 years

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